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INTRODUCTION

The renewed interest in advancing nuclear energy has spawned the research of advanced technologies for recycling nuclear fuel. A significant portion of the advanced fuel cycle includes the recovery of selected actinides by solvent extraction methods utilizing centrifugal contactors. Although the use of centrifugal contactors for solvent extraction is widely known, their operation is not without challenges. Solutions generated from spent fuel dissolution contain unknown quantities of undissolved solids. A majority of these solids will be removed via various methods of filtration. However, smaller particles are expected to carry through to downstream solvent extraction processes and equipment. In addition, solids/precipitates brought about by mechanical or chemical upsets are another potential area of concern. During processing, particulate captured in the rotor assembly by high centrifugal forces eventually forms a cake-like structure on the inner wall introducing balance problems and negatively affecting phase separations. One of the features recently developed for larger engineering scale Annular Centrifugal Contactors (ACCs) is the Clean-In-Place (CIP) capability. Engineered spray nozzles were installed into the hollow central rotor shaft in all four quadrants of the rotor assembly. This arrangement allows for a very convenient and effective method of solids removal from within the rotor assembly.

DESCRIPTION OF ACTUAL WORK

Prior to CIP testing, solids capture within the rotor were first evaluated by loading the rotor with a 0.1 weight percent solution containing diatomaceous earth in water. A test matrix varying flowrates from 1.9 to 11.4 lpm and rotor speeds from 1750 to 3500 rpm was used to evaluate solids capture.

Two procedures were used to evaluate the CIP operation. The first CIP procedure was conducted at a pressure of 40 psig and at a total flowrate of 25 lpm for 10 seconds. The second CIP procedure was conducted under identical conditions, but performed for 15 seconds. Each procedure was performed three times with approximately thirty seconds between each CIP operation.

A second challenge in contactor operation is the reliability of the unit itself. Reliability monitoring of an additional 12.5 cm contactor was initiated in April, 2006. This unit operates at 1750 rpm on a continuous basis. It was filled with approximately 4 liters of tap water and the heavy phase discharge hose was connected to one of the housing inlets. Therefore, the contactor is pumping at a full rate of about 20 liters per minute under recirculation. A manometer has been installed to monitor the liquid level in the mixing annulus and more water is added as needed to maintain level. Temperature, vibration and amperage draw are also monitored and recorded on a regular basis.

SUMMARY

Capture data indicate that removal efficiencies increase as rotor speeds increase. In addition, data indicate that as flowrates increase, the percent capture decreases. Capture % is dependant on both flowrate and rpm at the parameters tested. For all series tested, greater than 95% solids were captured by the contactor.

CIP processes performed at 40 psig (25.4 lpm) for three 15 or three 10 second cycles were very effective at the removal of solids from internal contactor surfaces.

To date, reliability measurements have provided no indication that the reliability of the unit is in question after one year of continuous operation.

I. INTRODUCTION

Testing with engineering scale ACCs has been ongoing at the Idaho National Laboratory. Prior testing includes hydraulic and mass transfer efficiency evaluations using commercially available contactors with rotor diameters of 5 cm and 12.5 cm. (1,2).

Solutions generated from spent fuel dissolution contain unknown quantities of undissolved solids. A majority of these solids will be removed via various methods of filtration prior to processing. However, smaller particles are expected to carry through to downstream solvent extraction processes and equipment. It is likely that the removal of these smaller solids will be captured and removed through centrifugal separation, (i.e, ACCs). Other potential areas of solids introduction include mechanical and chemical process (precipitation) upsets where solids can be captured within the rotor assemblies. This testing will evaluate the CIP capability to remove undesired solids present within the process. The Advanced Fuel Cycle Initiative (AFCI) has a goal to evaluate engineering scale ACCs for future aqueous-based spent nuclear fuel reprocessing.

One of the features recently developed for larger engineering scale ACC's is the clean-in-place (CIP) capability. During the design and testing of these contactors for separating crude oil from produced water, solids accumulated on the internal surfaces of the rotor requiring disassembly for cleaning. Cleaning by disassembly was time consuming, labor intensive, and prone to seal and bearing damage through mishandling. Therefore, a more convenient method of cleaning solids from the rotor was implemented in commercial ACCs with rotor diameters of 12.5 cm and larger (3, 4). Engineered spray nozzles were installed into the hollow central rotor shaft. A rotary union connects the source of the desired wash fluid for CIP to the rotor. CIP flushing is done when the ACC is offline and drained to provide a clear spray path to the inside diameter rotor wall and upper weir area. A view of the CIP design is presented in Figure 1.

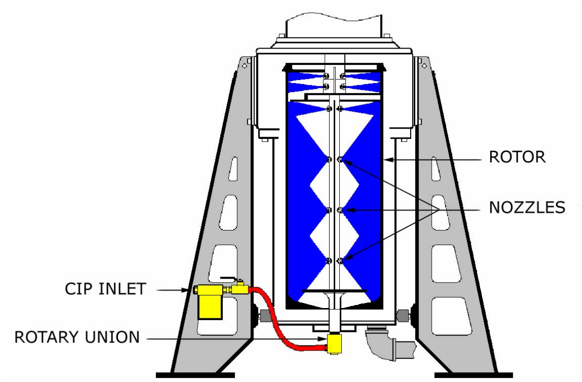

Fig. 1. Commercial contactor Clean-In-Place (CIP) design.

II. EXPERIMENTAL

II.A. Experimental Set-Up

The experimental test assembly consists of a single CINC V-5, 12.5 cm rotor diameter contactor fitted with a 2.550 inch heavy phase weir. A diatomaceous earth/feed slurry, contained in a 114 liter poly tank, was mixed continually using a Cole-Parmer®, Stir-Pak® laboratory mixer and was transferred to the contactor via a Seepex® progressive cavity pump (BN#2). Effluent exiting the contactor was collected in an additional 114 liter poly tank. The pump used to transfer cleaning solution to the CIP system was a Gould G&L series centrifugal pump. CIP pressure is monitored with a pressure gauge situated between the rotary coupling and CIP pump. This arrangement is capable of producing pressures of 30-60 psig with measured flowrates ranging from 13.2 to 27.9 lpm. The system is connected with industrial hose, cam and groove adapters, couplers, various pipe and Swagelok® fittings.

The CIP process includes halting the rotor and feed influent and draining holdup within the annulus and rotor. Draining process solution from the contactor exposes the internal rotor surfaces to the high pressure spray nozzles. Cleaning solution is pumped through the system via a permanently connected rotary coupling. After sufficient cleaning, the system is put back into service. The CIP process requires no disassembly/reassembly of the contactor, connections, or supply lines.

II.B. Capture Testing and Evaluation

A test matrix with varying flowrates from 1.9 to 11.4 lpm and rotor speeds from 1750 to 3500 rpm was used to evaluate solids capture within the rotor. Table 1 displays the test matrix used to evaluate solids capture. During testing, test numbers 4 and 5 could not be performed due to mechanical issues experienced with the progressive cavity pump. Test number 4, 9 and 14 are duplicate runs of tests 3, 8 and 13, respectively.

A 0.1 weight percent solution containing diatomaceous earth in water (mean particle size 80 micron and a density of about 2.4g/cc) was prepared as the surrogate feed slurry. Prior to sample collection for test #1, the feed solution was pumped through the contactor for 2 minutes to allow solution to fill the rotor and exit the contactor. Each test series was performed for approximately 1-3 minutes providing adequate time to reach equilibrium and obtain a four liter effluent sample. A single feed sample was obtained prior to tests 1-10 with second and third feed samples taken prior to tests 11-15.

TABLE I. Test matrix to evaluate solids capture within the rotor.

Test #	Desired Flowrate (lpm)	Measured Flowrate (lpm)	Rotor Speed (rpm)
1	1.9	1.5	1750
2	1.9	1.1	2300
3	1.9	1.9	2900
4	1.9	0*	2900
5	1.9	0*	3500
6	5.7	5.7	1750
7	5.7	5.7	2300
8	5.7	6.1	2900
9	5.7	5.7	2900
10	5.7	5.7	3500
11	11.4	11.4	1750
12	11.4	11.4	2300
13	11.4	11.0	2900
14	11.4	10.2	2900
15	11.4	11.0	3500

*pump failure at this flowrate

Four liter samples collected during testing were vacuum filtered through Nalgene® 0.2 micron (CN) disposable filters. Since the mass of solids present in the contactor effluent was very low, four liter samples were needed to provide an adequate mass of solids to determine removal efficiencies. Filters containing solids were dried in a StableTemp® laboratory oven at 50 ºC. Filters were allowed to dry for 3 days and were then weighed and recorded. Filters were then allowed to dry for an additional 2 days and were once again weighed and recorded. Five days of solids drying time was found to be sufficient to achieve stable weights for all samples. The percent capture was calculated using the following formula.

$$\%\,Capture = \frac{Solids\,Concentraton_{Feed} - Solids\,Concentraton_{sample}}{Solids\,Concentraton_{Feed}} \times 100\%$$

II.C. Clean-In-Place Evaluation

CIP testing was initiated by first loading the rotor with slurry solution remaining from the solids capture evaluation (~0.085 wt%). Slurry solution was transferred to the contactor at 7.5 lpm with the contactor operating at 2300 rpm. Approximately 109 liters of slurry solution was processed through the contactor. Once slurry transfer was complete, the rotor was stopped and allowed to slowly drain to avoid disrupting the solids cake within the rotor. After the rotor was completely drained of holdup solution, the contactor was dismantled to visually evaluate the presence of solids. Photographs were taken of various areas within the contactor showing solids buildup and location before careful reassembly.

Following observations, the contactor parts were cleaned of all solids. Solids were collected within the feed vessel and re-suspended for additional testing. The contactor was then reassembled and prepared for loading. Adhering to the previous procedure, slurry solution was transferred to the contactor at 7.5 lpm with the contactor operating at 2300 rpm. Approximately 109 liters of a ~0.085 weight percent slurry solution was processed through the contactor. Feed solution to the contactor was then halted and the rotor was stopped. Next, the drain was opened and holdup was drained from the system.

The first CIP test procedure was performed using a pressure of 40 psig at a total flow of 25 lpm for 10 seconds. This procedure was performed three times with approximately thirty seconds between each CIP operation. This procedure allowed the rotor to efficiently drain while internal rotor surfaces were cleaned. Four liter samples of CIP effluent were collected from the drain during the second and third CIP cycles. Solids were filtered under vacuum through 0.2 micron Nalgene® (CN) disposable filters and dried in a StableTemp® laboratory oven at 50 ºC. Filters were allowed to dry for three days and then weighed and recorded. Weighed filters were then placed in the oven for an additional 2 days and once again weighed and recorded. A second CIP test procedure was performed using a pressure of 40 psig at a total flowrate of 25 lpm for 15 seconds. This procedure was also performed three times with approximately thirty seconds between each CIP operation. In addition, four liter samples were also obtained during the second and third CIP cycles with samples being filtered, dried and weighed in the same fashion as the previous test procedure.

Once the CIP test procedures were complete, the contactor was dismantled and once again visually inspected for solids. Again, photographs of the cleaned contactor parts were taken.

II.D. Reliability Testing

The reliability of process equipment is of the utmost importance, especially when processing solutions containing radioactive constituents. Process shutdowns are both costly and can contribute to excessive personnel dose uptakes. The reliability testing of CINC V-05 contactors provides process design engineers with information necessary to support full scale implementation.

Reliability monitoring of an additional 12.5 cm contactor was initiated in April, 2006. This unit operates at 1750 rpm on a continuous basis. It was filled with approximately 4 liters of tap water and the heavy phase discharge hose was connected to one of the housing inlets. Therefore, the contactor is pumping at a full rate of about 20 liters per minute under recirculation. A manometer was installed to monitor the liquid level in the mixing annulus and more water is added as needed to maintain level. During operation, vibration measurements are obtained using a Balmac™ (model 205) hand-held vibration meter. Measurements were obtained from startup and taken daily for one week. Once daily measurements were completed, measurements were then taken weekly. Four locations on the CINC V-05 were chosen to measure vibration and were appropriately labeled A, B, C and D. Location A is located at the bottom of the motor housing near the motor/contactor coupler housing. Locations B and C are situated at the top and bottom bearing flanges. Location D is located directly on the CIP rotary coupling.

In addition to vibration measurements, temperature readings are also taken. Temperature was measured using a Fluke thermometer (model 52) utilizing a Type-K thermocouple. Locations for temperature readings are nearly identical to those of vibration. However, location C is situated on the contactor housing above the lower bearing flange. Locations A, B and D are identical.

Amperage draw values are also obtained from the frequency drive controller (Allen Bradley Powerflex 4). Following power interruptions, the unit is restarted as soon as discovered and outages logged. A photo of the CINC V-05 during reliability testing is shown in Figure 2.

Figure 2. A photo of the CINC V-05 during reliability testing.

III. RESULTS

III.A. Capture Testing and Evaluation

Table 2 provides the percent capture data for the capture evaluation. Figure 2 displays the percent capture as a function of rpm and flowrate. As was expected, data indicate that capture efficiencies increase as rotor speeds increase. This observation is most prevalent in the series at the 11.4 lpm flowrate. For example, the percent capture at 1750 rpm was 95.9%, but at 2900 rpm the percent capture increased to 98.2%. The percent capture at 2900 and 3500 rpm were 99.1 and 99.4%, respectively.

The data also indicates that as flowrates increase at constant rpm, the percent capture decreases. This is due to the higher residence time associated with lower flowrates. The 1750 rpm series displays the greatest decrease in capture with increasing flowrate followed by the 2300, 2900 and 3500 rpm series. These data demonstrate that percent capture is impacted by both flowrate and rotor speed.

TABLE II. Percent capture data for the capture evaluation.

Test #	Measured Flowrate (lpm)	Volume Filtered (mLs)	Average (wt%)	Average Capture (%)
Feed #1		4180	0.09582	
Feed #2		4320	0.08381	
Feed #3		4088	0.08560	
1	1.5	3935	0.00016	99.8
2	1.1	3838	0.00010	99.9
3	1.9	3880	0.00010	99.9
4	0*	n/a	n/a	n/a
5	0*	n/a	n/a	n/a
6	5.7	3956	0.00131	98.6
7	5.7	3869	0.00063	99.3
8	6.1	3711	0.00029	99.7
9	5.7	3905	0.00031	99.7
10	5.7	3870	0.00022	99.8
11	11.4	3651	0.00052	99.4
12	11.4	3962	0.00150	98.2
13	11.0	3962	0.00073	99.1
14	10.2	3949	0.00071	99.2
15	11.0	3875	0.00347	95.9

*pump failure at this flowrate

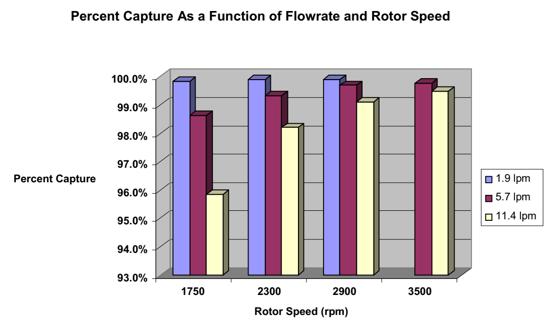

Figure 3. Percent capture as a function of flowrate and rotor speed.

III.B. Clean-In-Place Evaluation

Figures 4 through 7 pictorially display the presence and location of solids accumulation within the contactor assembly following loading of 109 L of a 0.1 wt % diatomaceous earth solution. Figure 4 displays a thick cake of diatomaceous earth within the rotor. It should be noted that an evenly distributed cake was most likely present during operation, but during rotor holdup draining, the solids sloughed to the bottom of the rotor assembly. Figure 5 displays solids present in the upper weir assembly. Though difficult to see, minimal solids were present in the upper weir package indicating a sufficient cake had formed allowing solids to pass over the upper weir and exiting the contactor with the effluent. When referring to figure 6, solids can be observed in the bottom of the vane package (top center of photo). Figure 7 displays solids present in the bottom mixing plate.

Fig. 4. Solids capture, inside rotor.

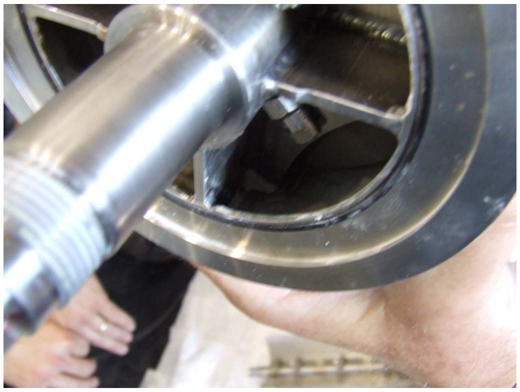

Fig. 5. Solids capture, upper weir.

Fig. 6. Solids capture, vane package.

Fig. 7. Solids capture, mixing plate.

Solids recovered from the second and third CIP cycles were measured to determine the CIP efficiency. It should be noted that the majority of solids were flushed from the system during the first CIP cycle. As a result, samples of the first CIP effluent were not obtained.

Table III. CIP efficiency as determined by CIP effluent solids recovery.

CIP #	Sample Vol (Liters)	Recovered Solids Wt. (g)	CIP Efficiency (%)
CIP-1, #2	4.00	2.751	97
CIP-1, #3	3.85	0.015	> 99
CIP-2, #2	4.11	2.854	97
CIP-2, #3	4.15	0.015	> 99

With a feed solution measured at 0.085 weight percent diatomaceous earth, it was calculated that a total 92.65 grams of solids remained in solution. Solids recovered from the CIP-1, #2 and #3 cycles displayed CIP efficiencies of 97 and >99%. Solids recovered from the CIP-2, #2 and #3 cycles also displayed CIP efficiencies of 97 and >99%.

Solids recovered for each CIP cycle are in excellent agreement for both tests. This agreement would indicate that both 10 and 15 second CIP cycles provided very similar cleaning. Solids recovered from the third CIP sample, from each test, indicates that there were minimal solids remaining in the rotor. Three CIP cycles following the initial rotor draining sequence provided nearly complete removal of captured solids from internal contactor surfaces.

Figures 8 through 11 pictorially summarize the complete removal of solids within the contactor assembly following the ten second CIP clean out test. Figures 8 and 10 display the absence of solids within the rotor and vane packages. In addition, no solids were present in the upper weir or bottom mixing plate as shown in figures 9 and 11. Pictures of contactor parts following the second CIP process performed for 15 seconds are not shown. However, comparable results were obtained.

Fig. 8. Following CIP, inside rotor.

Fig. 9. Following CIP, upper weir package.

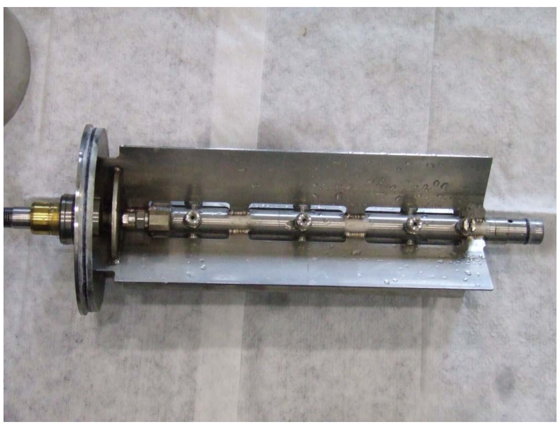

Fig. 10. Following CIP, complete vane package.

Fig. 11. Following CIP, mixing plate.

The CIP process, regardless of pulse time, was very effective at the removal of solids from internal contactor surfaces. CIP times greater than thirty seconds may partially fill the rotor with cleaning solution and obstruct internal rotor surfaces to the full effects of the high-pressure spray nozzles. Although the CIP effluent failed to exit the open inlet following a 30 second CIP time, it is recommended that three 10 or 15 second CIP cycles should be performed.

III.C. Reliability Testing

Over the 14 month period there has been no indication from trends in vibration, temperature or amperage draw that would reflect changes to the operational reliability of the contactor are occurring. Reliability testing is on going.

IV. CONCLUSIONS

A test matrix varying flowrate from 1.9 to 11.4 lpm and rotor speeds from 1750 to 3500 rpm was used in capture testing and CIP evaluation. Capture data indicate that removal efficiencies increase as rotor speeds increase. In addition, data indicate that as flowrates increase, the percent capture decreases. Capture % is dependant on both flowrate and rpm at parameters tested. For all series tested, greater than 95% solids were captured by the contactor. The diatomaceous surrogate used is about 72% of the density calculated for the spent fuel undissolved solids, assuming all are in oxide form. Therefore, one would expect that the ACC capture efficiency may improve when processing actual dissolved fuel solutions.

CIP processes performed at 40 psig (25.4 lpm) for three 15 or three 10 second cycles were very effective at the removal of solids from internal contactor surfaces In addition, previous experience has shown some solids holdup within the torturous path of the bottom housing mixing plate. Additional drains could be added to alleviate this potential problem and should be considered in future modifications.

To date, reliability measurements have provided no indication that the reliability of the unit is in question after more than a year of continuous operation.

V. REFERENCES

1. D.H. Meikrantz, J.D. Law, R.S. Herbst, T.G. Garn, N.R. Mann, T.A. Todd, “Hydraulic and Mass Transfer Testing of Commercial 5 cm Annular Centrifugal Contactors at INL”, INL/EXT-05-00793, October 2005

2. D. H. Meikrantz, T.G. Garn, N.R. Mann, J.D. Law, T.A. Todd, “Low Mix Testing of 5 cm Annular Centrifugal Contactors”, INL/EXT-06-11058, March 2006

3. U.S. Patent 5,908,376, “Self Cleaning Rotor for a Centrifugal Separator”, Jun. 1, 1999.

4. Meikrantz, D.H., Meikrantz, S.B., Macaluso, L.L., “Annular Centrifugal Contactors for Multiple Stage Extraction Processes”, Chem. Eng. Comm., 188, 115-127, 2000.